

## REMARKS

By this Amendment, claims 1, 3 and 5-7 have been amended, claims 2 and 4 have been canceled and new claims 9 and 10 have been added to the application. Accordingly, claims 1, 3 and 5-10 are pending in the application. No new matter has been added.

In the prior Office Action, the Examiner rejected claims 1-8 under 35 U.S.C. §102(b) as being anticipated by Mellul, U.S. Pat. 5,612,021. The Examiner contends that Mellul teaches a cosmetic composition that includes a wax, a resin and a hollow powder. Applicants respectfully disagree.

Mellul discloses the use of fullerenes as pigments in cosmetic compositions (see 1, lines 52-55). The Examiner equates fullerenes with the "hollow powders" claimed by applicants. Applicants respectfully submit that this is an improper conclusion.

While it is true that fullerenes are completely closed hollow spheres of carbon atoms, fullerenes cannot be considered as "hollow powders" as claimed in claim 1. Fullerenes are "hollow" only at the atomic level. The inner "hollow" portion of a fullerene molecule usually defines a vacuum. Only after extraordinary processing can any molecules be forced into the "hollow" of a fullerene molecule, and even then in rare cases (see <http://xbeams.chem.yale.edu/~cross/fullerene.html>, Fullerene Research in the Cross Group, Yale University, Department of Chemistry, wherein it is reported that a noble gas atom can only be forced into the "hollow" of about 0.1% of fullerene molecules) (copy of web page is attached). Thus, fullerenes cannot be fairly characterized as "hollow powders" as claimed in claim 1.

In addition, Mellul cannot be said to fairly disclose an eyelash cosmetic that includes all three of the elements presently claimed in claim 1 in the amounts claimed in claim 1. Mellul discloses many different types of cosmetic compositions that can include fullerenes as pigments. In Example 1, Mellul discloses a mascara composition. However, while the mascara composition according to Mellul does include a wax, it clearly does not include a resin, and further does not include a hollow powder. In an attempt to overcome this deficiency, the Examiner refers to col. 7, lines 1-5 wherein Mellul discloses that certain cosmetic compositions may comprise "resins". But this

teaching is in the context of "nail varnishes", and not eyelash cosmetics. Mellul simply does not teach or suggest an eyelash cosmetic that includes (a) a wax, (b) a resin and (c) a hollow powder in the amounts claimed in claim 1. And one of ordinary skill in the art in view of the teachings of Mellul would not be motivated to create an eyelash cosmetic as claimed in claim 1.

As noted above, applicants have added new claims 9 and 10 to round out applicants' claim coverage. New claim 9 specifies that the hollow powder is a hollow resin powder as disclosed on page 6, paragraph [0015] through page 7, paragraph [0017] of the specification. And new claim 10 specifies that the hollow powder is a hollow glass powder as disclosed on page 7, paragraph [0018] through page 8, paragraph [0019] of the specification. Clearly, claims 9 and 10 add no new matter to the application, and further differentiate the hollow powders as claimed from the fullerenes disclosed in Mellul.

In light of the foregoing, it is respectfully submitted that the present application is in a condition for allowance and notice to that effect is hereby requested. If it is determined that the application is not in a condition for allowance, the Examiner is invited to initiate a telephone interview with the undersigned attorney to expedite prosecution of the present application.

If there are any additional fees resulting from this communication, please charge the same to Deposit Account No. 18-0160, Order No. IWI-16232.

Respectfully submitted,

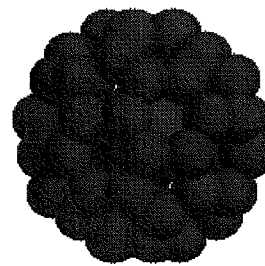
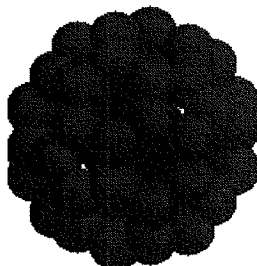
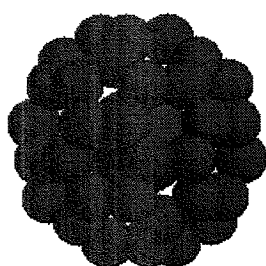
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# Fullerene Research in the Cross Group

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In collaboration with Prof. Martin Saunders we make fullerenes with atoms and small molecules trapped inside and study their properties. These are van der Waals molecules in that there is no chemical bond between the trapped atom or molecule and the carbon cage. Yet, they are very stable, since the atom cannot escape unless several bonds are broken. Numbers refer to publications listed below. So far we have put He, Ne, Ar, Kr, Xe and tritium and nitrogen atoms, as well as He<sub>2</sub>, Ne<sub>2</sub>, CO, and N<sub>2</sub> inside a variety of fullerenes. The tritium atoms are inserted by generating them at high energies in a nuclear reaction. The noble gases are put in by heating the fullerenes in the presence of the gas at high temperatures and pressures or by shooting them in as ions or metastable atoms. The noble gas compounds can be detected by mass spectroscopy either as intact molecules or by decomposing them and detecting the noble gas. In the case of <sup>3</sup>He we can see the <sup>3</sup>He NMR signal.

The chief method used to make the noble-gas fullerene compounds is to heat fullerene in the presence of the gas at 650°C and 3000 atm.[3] We make an ampoule from a tube of OFHC copper by crimping one end, filling it, and crimping off the top. The ampoule is placed in a high-pressure bomb which is then filled with water, closed and heated to 650°. The pressure rises to 3000 atm, and the copper ampoule is squashed flat, compressing the gas within. After about 8 hrs., the bomb is cooled, and the ampoule is opened. The fullerene is extracted in CS<sub>2</sub>. About 85% of the fullerene is soluble, and about 0.1% of the molecules contain a noble gas atom. In the cases of He and Ne, we find small amounts of C<sub>70</sub> containing two helium atoms[20] or two neon atoms [18]. We have found even smaller amounts of C<sub>60</sub> containing two helium atoms[37].

Tritium atoms with high kinetic energy are generated using a nuclear reaction.[6][26] We prepare either a lithium salt of C<sub>60</sub> or use <sup>3</sup>He@C<sub>60</sub> or a mixture of C<sub>60</sub> and <sup>3</sup>He gas. <sup>6</sup>Li and <sup>3</sup>He each absorb thermal neutrons in a reactor to give tritium. The tritium then loses energy by ionizing the fullerene until it eventually stops. Some of the time it stops inside a fullerene molecule which then remains stable. We can isolate tritium labeled C<sub>60</sub>. In the case of the T@C<sub>60</sub> generated from lithium, we obtain trace amounts of <sup>3</sup>He@C<sub>60</sub> formed by the radioactive decay of the tritium. If the tritium were on the outside, the <sup>3</sup>He would be on the outside and would be lost.

We have constructed a beam machine to put atoms inside fullerenes.[15] In the center is a cylindrical target, rotating slowly. On one side is an oven which produces a continuous beam of fullerene. Thus we have a freshly deposited surface of fullerene on the target. On the other side is a source of noble gas ions or metastable neutrals which hit the surface. The ions and metastables are made in an electric discharge. Ions are extracted by an electric field and bent by 90°. The amount of incorporation for He<sup>+</sup> is small at 30eV and rises to a maximum near 100eV and then decreases. Above 100eV the fullerene is partially destroyed. For Ne<sup>+</sup>, the yield is smaller, and the threshold is about 100eV. In the metastable mode, the ions are bent away from the target, while the metastables hit it. We find incorporation of both He\* and Ne\*. The method also works for nitrogen atoms. N@C<sub>60</sub> consists of a free nitrogen atom with three unpaired electrons unbound to the carbon cage. It gives a clean atomic-like ESR signal. We have used the beam method to put He into dodecahedrane, C<sub>20</sub>H<sub>20</sub>, a hydrocarbon cage. [24]

<sup>3</sup>He labeled fullerenes and their derivatives can be studied by NMR spectroscopy.[2][10] The pi electrons around the fullerene molecules cause large diamagnetic shielding and an upfield shift of the <sup>3</sup>He line relative to dissolved <sup>3</sup>He gas. C<sub>60</sub>

has an upfield shift of 6.4 ppm and  $C_{70}$  28 ppm. Higher fullerenes fall between these limits.[8][25] Adding groups to the outside changes the pi electron structure and the chemical shift of the  $^3\text{He}$ . [4][5][7][10][47] The most common adduct is across one of the 6,6 double bond joining two hexagons. Single addition usually causes an upfield shift of about 3 ppm from  $C_{60}$ , the exact amount depends on the group being added. Addition across a 5,6 single bond joining a pentagon and a hexagon gives a much smaller shift. Multiple additions give a more complicated picture.[14] Each fullerene molecule and each fullerene adduct gives a different, unique NMR line. Adding six electrons to  $C_{60}$  and to  $C_{70}$  gives another closed-shell species. The anions have very different chemical shifts from the neutrals.[21] We also see the NMR signal for  $^{129}\text{Xe}$  in  $\text{Xe}@C_{60}$ . The NMR of  $^{129}\text{Xe}@C_{60}$  is different from that of  $^3\text{He}@C_{60}$ . [35]

We have constructed a mass spectrometer to analyse the noble gas inside the fullerenes.[12] We find that very pure  $C_{60}$  is extraordinarily stable. At 630°C the half life for decomposition is greater than one month, but even trace quantities of solvent or air absorbed in it will catalyze its decomposition. At 900°C the half life is 10 hours. In both cases the gas is largely or completely released by the decomposition of the  $C_{60}$ . Using a different mass spectrometer, we can directly see the peaks for the various compounds.

Using multiple passes through an HPLC column gave us nearly pure  $\text{Kr}@C_{60}$ . [22] We could see the small shift in the  $^{13}\text{C}$  NMR due to the presence of the Kr atom. There were small shifts in the IR, visible, and UV lines and a 10% decrease in the life time of the lowest triplet state as well. A similar separation has been achieved with  $\text{Xe}@C_{60}$ . [35]

Using classical statistical mechanics, we can calculate the equilibrium constant for the incorporation of a noble gas atom into  $C_{60}$ . [11] We start with a potential function  $V(R)$  for the gas atom as a function of the distance from the center.  $V(R)$  is obtained by using one of several literature potentials between the gas atom and each carbon atom. We can then calculate the equilibrium constant using classical statistical mechanics  $q^*$  is the usual partition function for  $\text{X}@C_{60}$  not including the gas motion,  $q_{int}$  is the internal partition function for  $C_{60}$ , and  $p_X$ . The values for He and Ne seem to be relatively independent of the potential model used, but the values for the higher noble gases are much more sensitive to the choice of potential. For He and Ne the equilibrium values are much higher than the amounts that we can get from our experiments, so that the experiments are far from reaching equilibrium.

9,10 dimethyl anthracene (DMA) reacts reversibly with  $C_{60}$ . By measuring the  $^3\text{He}$  NMR peak heights as a function of DMA concentration, we can get the equilibrium constants for the addition of successive DMA molecules to  $C_{60}$  and  $C_{70}$ . [28] By doing this as a function of temperature, we can get the enthalpy changes as well. We found that the various isomers of  $C_{84}$  have very different equilibrium constants for the addition of DMA, and this can be used as a basis for separation of the isomers. [25]

By adding suitable groups to the outside of fullerenes, it is possible to open a hole in the cage. We have measured both equilibrium constants and kinetics for noble gases entering and leaving chemically opened fullerenes. [31][44][50]

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